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ILIKON CORPORATION
NATICK, MASSACHUSETTS

BASIC STUDIES ON DISPERSION HARDENING

Robert Grierson

QUARTERLY REPORT

12 October - 11 January, 1965

Contract NASw-726

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APPROVED:

L. J. Bonis
Technical Director

February 10, 1965

QUARTERLY REPORT - PROJECT 211 - NASw-726

1. Introduction

During the quarter currently being reported (12 October, 1964 - 11 January, 1965), research has been carried out in two fields. These are:

1) The detection of interfacial strains, in internally oxidized alloys, using transmission electron microscopy.

2) The effect of annealing on the structure of cold worked internally oxidized alloys and in particular on TD nickel. This study has developed out of our attempts at investigating the interfacial strains in these alloys by means of x-ray diffraction.

2. Interfacial Strain Measurements

Two further nickel base alloys have been obtained. These are Ni + ½% Al and Ni + 1% Si. It is hoped to be able to internally oxidize the nickel-aluminum alloy under conditions such that very fine (<100 Å) particles will be obtained. As was explained in the previous quarterly report, these particles should be coherent with the matrix and it is therefore expected that the interfacial strains should be measurable by observation of the D shaped lobe, diffraction effect. The purpose of the nickel-silicon alloy is to allow us to observe interfacial strains which are produced on quenching nickel-silica alloys. Due to the comparatively large difference in coefficient of expansion between nickel and silica, interfacial strain should be more readily observable in this system. Initial experiments involving the internal oxidization of thin foils of Ni-Al and Ni-Si have been carried out and it is intended to internally oxidize and fabricate powders in the next few weeks.

3. The Effect of Annealing on Cold Worked TD Nickel

(a) Introduction

One of the most interesting features of the mechanical properties of dispersion strengthened alloys is their resistance to recovery from cold working. It has been observed that the existence of a stable dispersed phase within a matrix can raise, by several hundred degrees, the temperature at which recovery will occur. The existence of cold work involves the existence of strains, and we have decided to investigate the strains retained after annealing cold worked material in order to discover if they exist in the interfacial regions, in the matrix in general or at both the interface and in the matrix. The retention of cold work after annealing at temperatures up to the service temperature of a dispersion strengthened alloy would, of course, be a very important factor in determining its strength at its service temperature. For this reason, it was felt that, even if we did not find that the retained strains were interfacial in nature, valuable information would be obtained by the investigation currently being carried out.

The two techniques, which we developed earlier in this research program, have been used to investigate the retained cold work in these materials. These techniques are transmission electron microscopy and the Fourier analysis of the profile of x-ray diffraction lines.

(b) X-Ray Diffraction Measurements

As yet, the only results obtained have been qualitative in nature. Figure 1 compares the shape of the 200 line for pure nickel and TD nickel after each has been cold rolled to give an 80% reduction in thickness, and Figure 2 compares the same specimens after they have also been given a 1 hour anneal at 700°C. It can be seen that annealing causes a considerable change to occur in the line profile of the nickel specimen, while little change occurs in that of the TD nickel. The pure nickel is fully annealed and contains no cold work, while the nickel matrix of the TD nickel composite still contains considerable cold work.

Figure 3 shows the line profile obtained using similar TD nickel specimens which have been annealed for 5 hours at 900°C, and 1 hour at 1440°C. In neither case does the broadening of the diffraction line disappear. In both cases the broadening is still much greater than that observed for pure nickel after a 700°C anneal. It was found that prolonged annealing of TD nickel at any given temperature caused little if any change in the line shape as compared with that for a 1 hour anneal.

It would, therefore, appear that, even after annealing at temperatures within 15°C of the melting point of nickel, the nickel matrix of TD nickel is not in a fully annealed form. Attempts were made to anneal TD nickel within 5° of the nickel melting point - these caused partial melting of the sample and the material was therefore discarded.

These initial, qualitative x-ray results indicate a gradual increase in the amount of recovery, as measured by line breadth, which occurs as the annealing temperature is increased. They also indicate that complete recovery does not occur even on annealing at temperatures within 15°C of the melting point.

(c) Transmission Electron Microscopy

Transmission electron microscopy has been carried out on both TD nickel and pure (99.95%) nickel. Figures 4 and 5 show these materials after they have been cold rolled to give an 80% reduction in thickness. A heavy dislocation structure exists in each. Figures 6 and 7 show these same two materials after they have also received a one hour anneal at 700°C. The structure of the TD nickel is virtually unchanged whilst complete recovery of the pure nickel has occurred. Figures 8 and 9 show two areas of a TD nickel foil which has been annealed for 1 hour at 900°C. In Figure 8, no recovery is apparent, but in Figure 9 partial recovery has occurred. At 900°C recovery is just starting to occur - it does not occur uniformly throughout the sample but rather only in certain localized areas. Figures 10 and 11 show

TD nickel which has been annealed for 1 hour at 1100°C and 1440°C respectively. In these samples, the dislocation structure observed after annealing at lower temperatures is no longer present. The only structure observed, other than the dispersed phase particles, is the presence of twins. These are observed after annealing both at 1100°C and 1440°C.

The transmission electron microscopy results indicate that the major change in structure which occurs on increasing the annealing temperature does so in the region of a 900°C annealing temperature.

(d) Proposed Further Work

The next stage of this investigation is to carry out a Fourier Analysis of the profiles of the x-ray diffraction lines and thus obtain quantitative information about the way in which the internal structure is changing. These results will give us a fairly complete description of the internal structure of the material examined. Further transmission electron microscopy will then be carried out to check the validity of the x-ray results thus obtained.

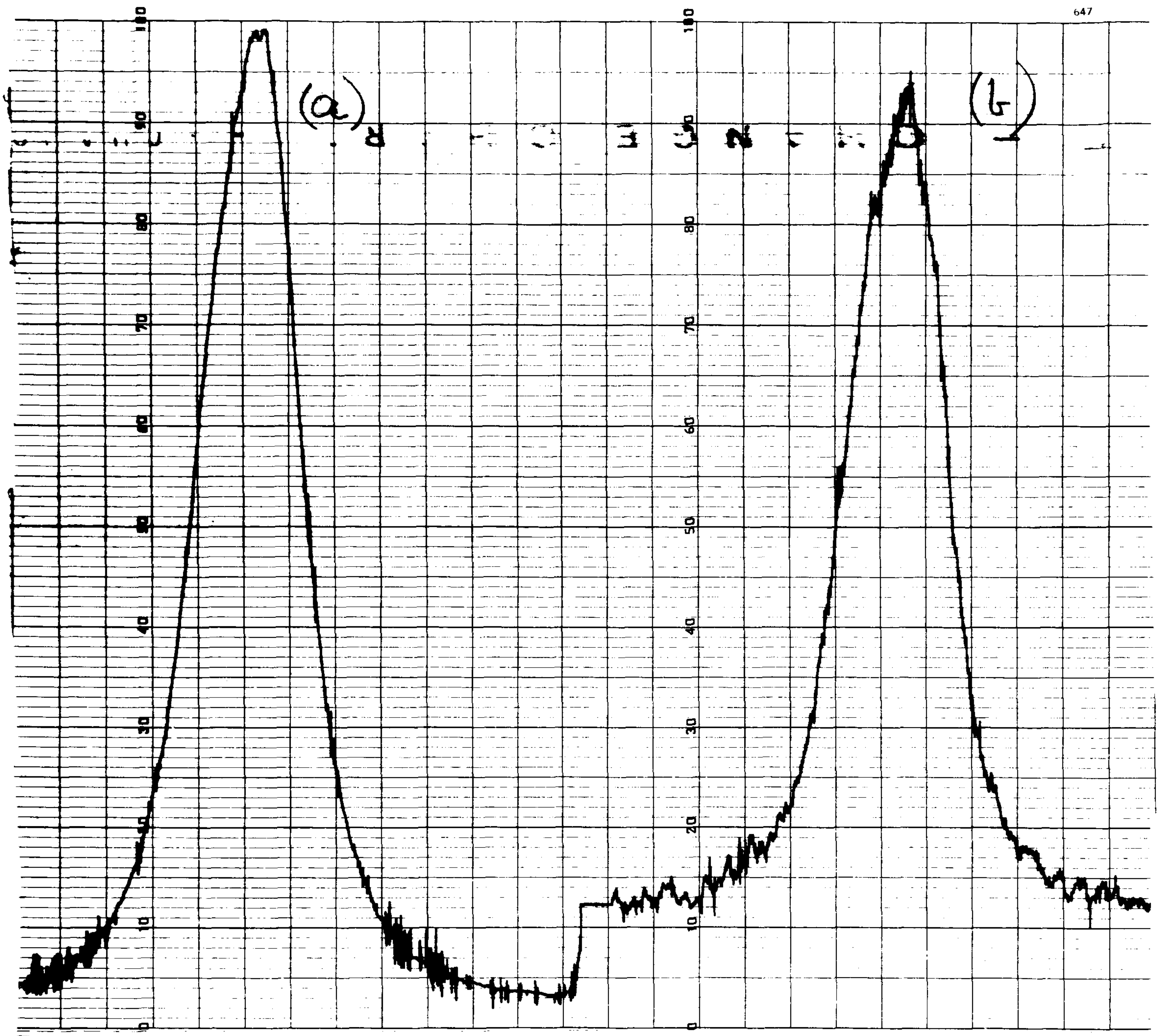

FIG. 1 The 200 x-ray diffraction lines for

(a) 80% cold worked TD nickel
(b) 80% cold worked pure nickel

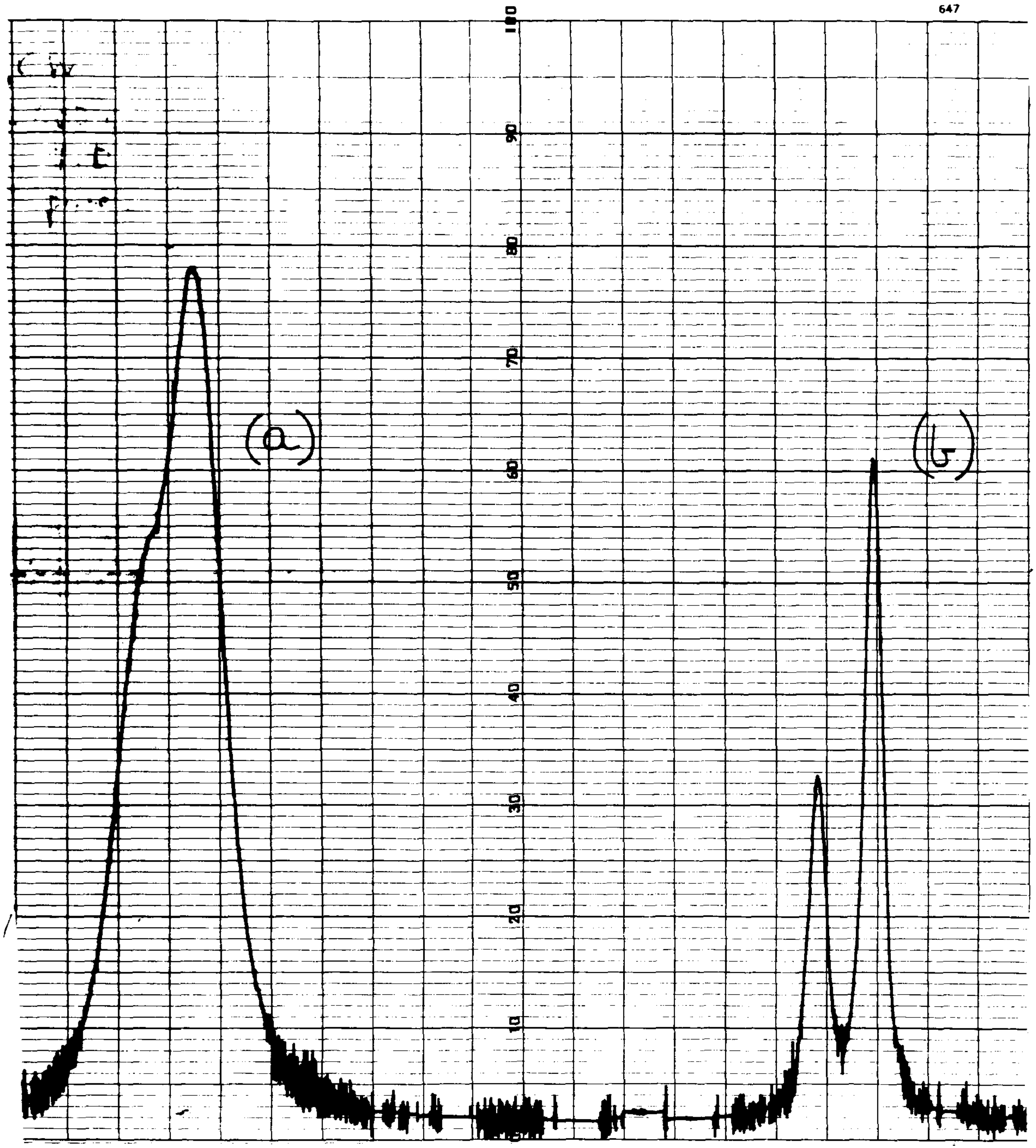

FIG. 2   The 200 x-ray diffraction lines for

(a) 80% cold worked TD nickel which has subsequently been annealed for 1 hour at $600^{\circ}C$

(b) 80% cold worked pure nickel which has subsequently been annealed for 1 hour at $600^{\circ}C$

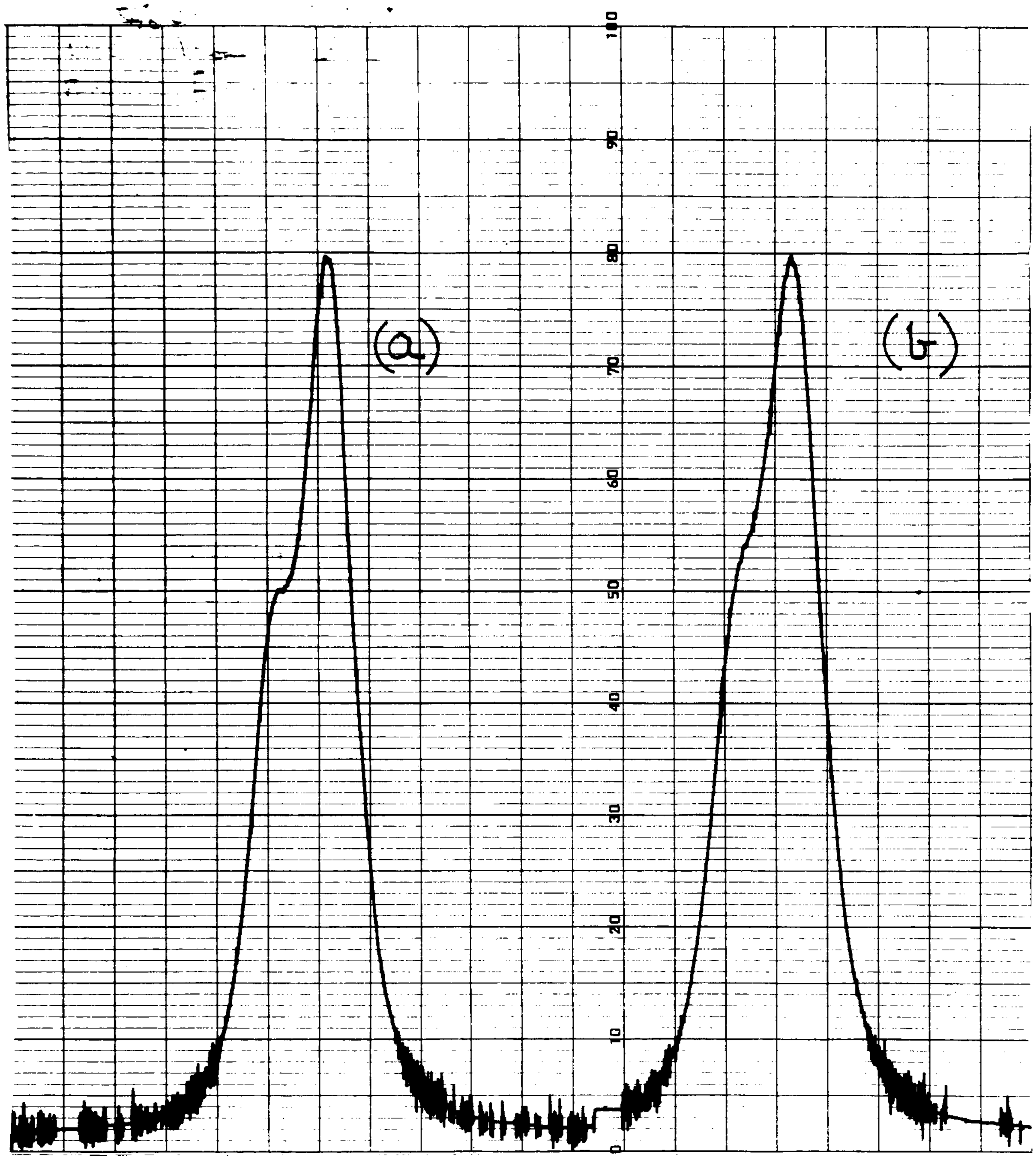

FIG. 3 The 200 x-ray diffraction lines for TD nickel which has been 80% cold worked and annealed for

(a) 1 hour at 1440°C
(b) 5 hours at 900°C

FIGURE 4 T. D. Nickel 80% Cold Worked

FIGURE 5 Pure Nickel 80% Cold Worked

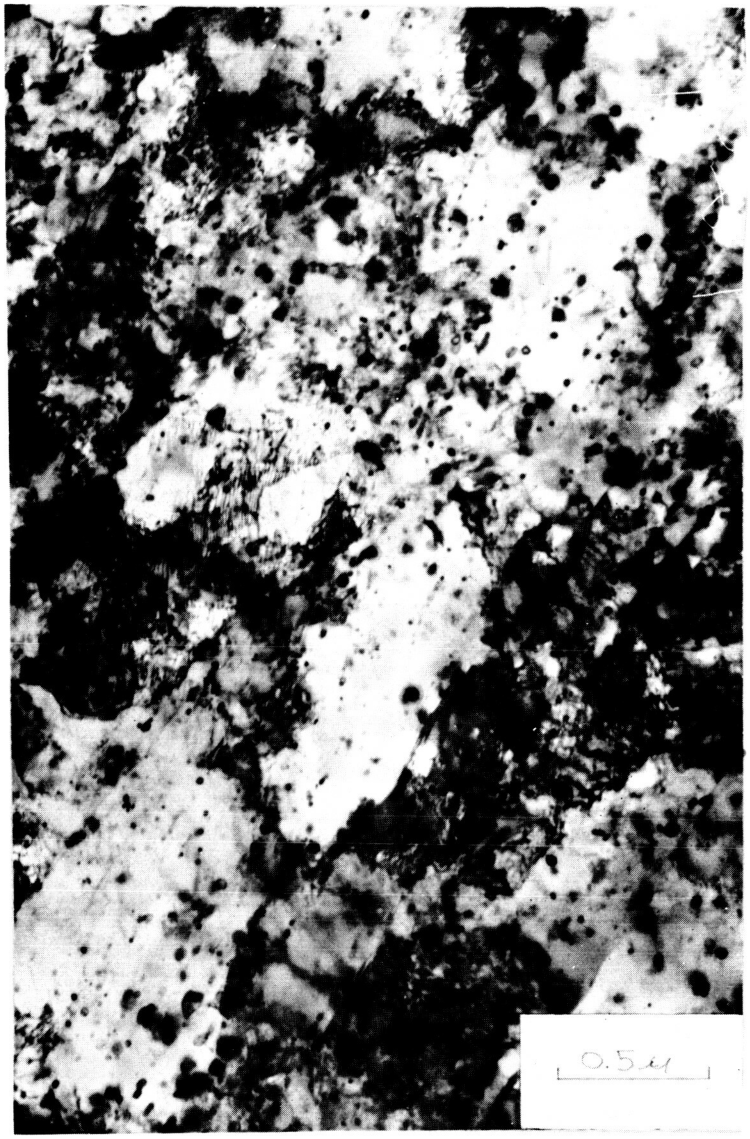

FIGURE 6 T. D. Nickel 80% Cold Worked then annealed for 1 hour at 700 °C

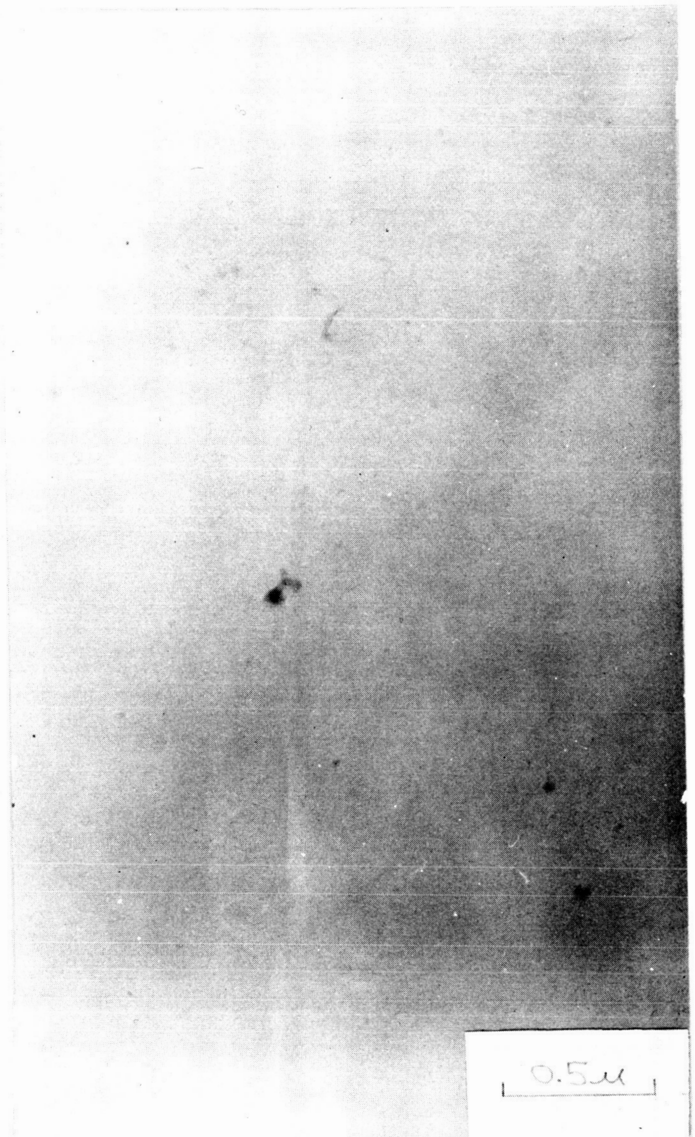

FIGURE 7   Pure Nickel 80% Cold Worked
then annealed for 1 hour at 700°C

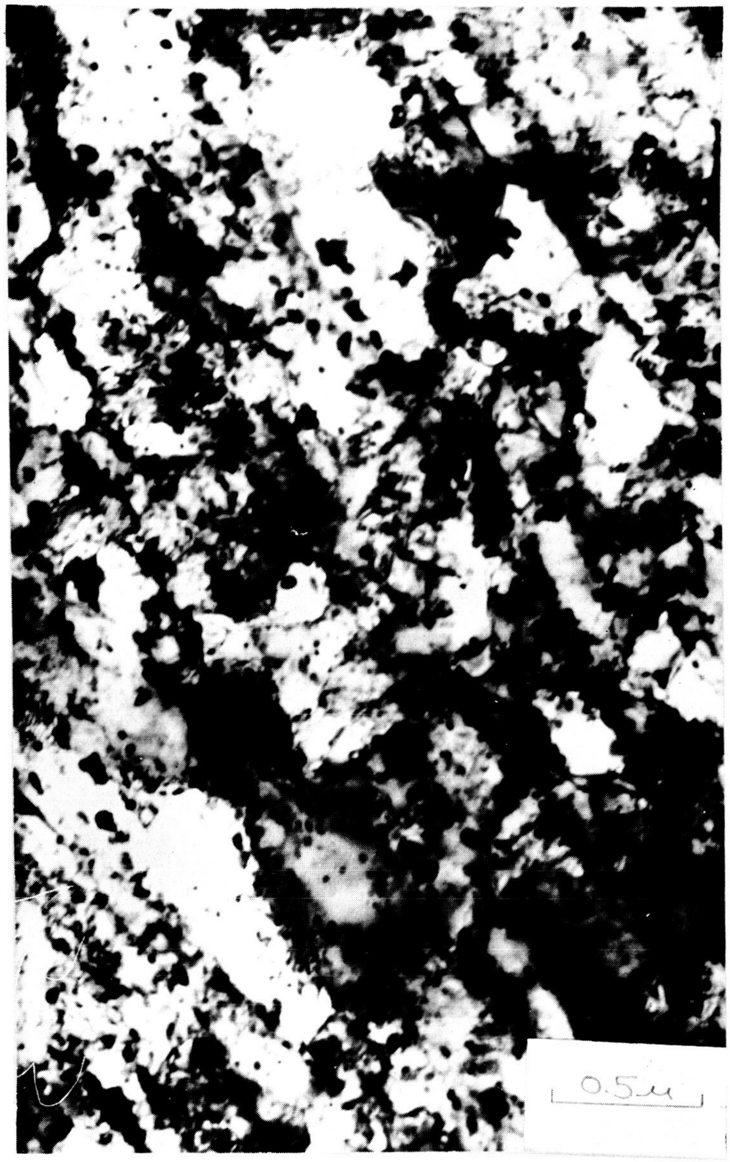

FIGURE 8 T. D. Nickel 80% Cold Worked then annealed for 1 hour at 900°C This specimen shows no obvious recovery

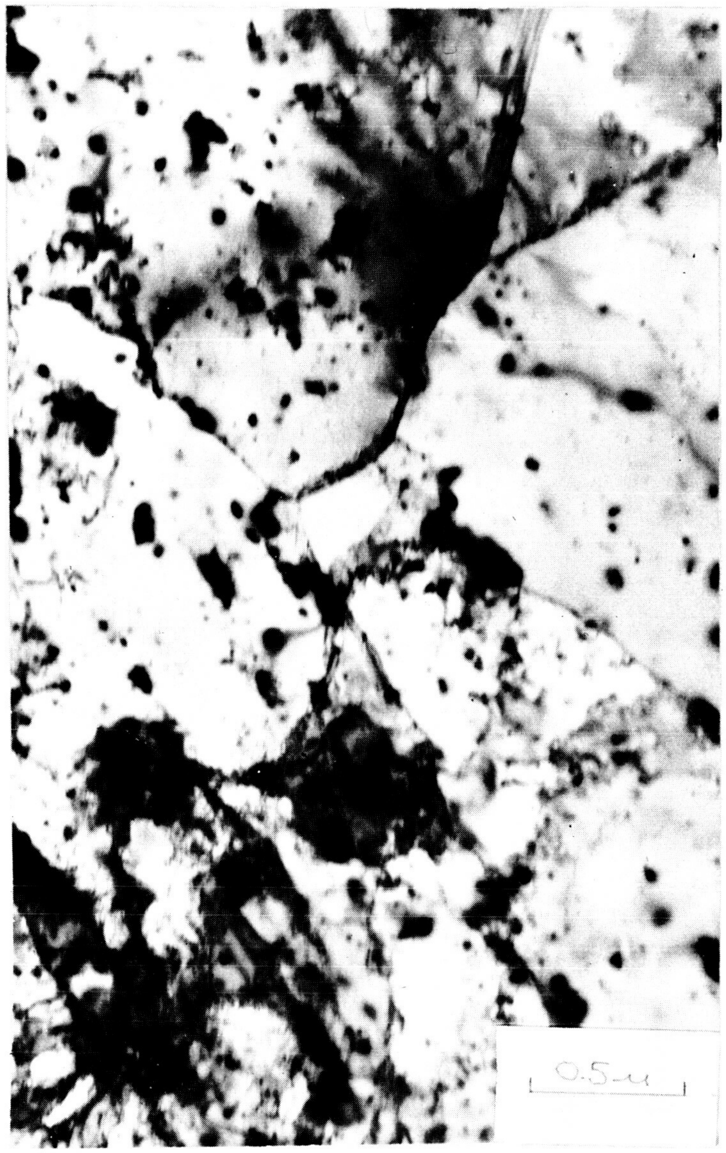

FIGURE 9 T. D. Nickel 80% Cold Worked then annealed for 1 hour at 900°C Partial recovery has occurred

FIGURE 10 T. D. Nickel 80% Cold Worked then annealed for 1 hour at 1100°C

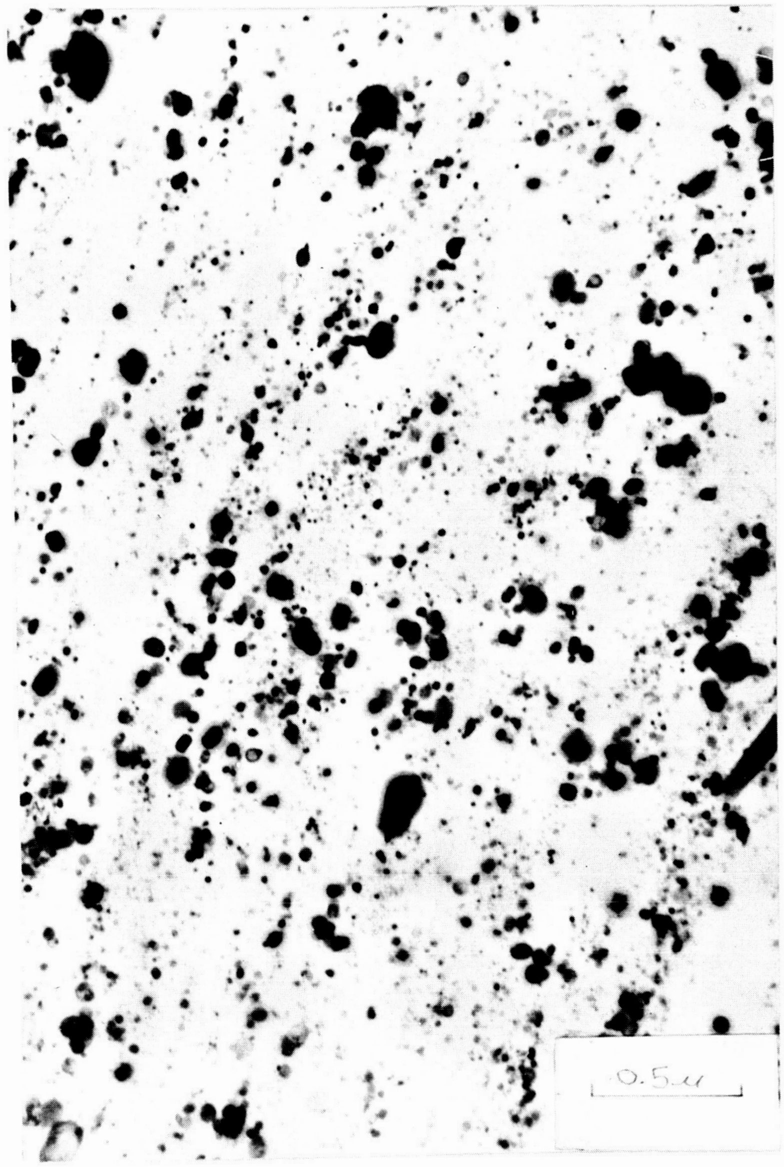

FIGURE 11 T. D. Nickel 80% Cold Worked then annealed for 1 hour at 1440 °C